

# Liquid–liquid extraction of sulphuric acid from zinc bleed stream

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## Abstract

Recovery of both sulfuric acid and zinc from the spent zinc bleed stream (SZBS) generated during the electrowinning of zinc in the zinc refineries using tris-(2-ethylhexyl) amine (TEHA) dissolved in kerosene has been investigated in detail. Extraction was studied under various experimental conditions such as concentration of TEHA in the organic phase and temperature, concentration of acid and zinc present in the initial aqueous feed, temperature of extraction, equilibrium time. Increasing the experimental temperature led to a decrease in the extraction of acid. The extraction behaviour of zinc present in the spent liquor was examined under the optimized conditions for its co-extraction with the acid. It was found that in the sulphate medium, zinc is not extracted along with the acid and the raffinate can be used for the preparation of zinc oxide. Thus under the optimised conditions acid extraction was found to be 182 g/L of H<sub>2</sub>SO<sub>4</sub> with 75% TEHA in 3–5 min of phase contact. Extraction increases with the increase in O/A ratio and was found to be about 94% at O/A of 5 with the initial feed concentration of 173.5 g/L of sulphuric acid. After extraction process, stripping was performed to recover the loaded acid and regenerate the solvent for recycling. Resultant regenerated organic phases can be reused in succeeding extraction step with the yield ranging from 94.8% to 99.7% depending on composition of the initial feed solution. Effect of temperature was studied at 30, 45 and 60 °C. The effect of the number of extraction and stripping stages and the volume phase ratios in extraction and stripping upon sulphuric acid recovery is discussed.

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## 1. Introduction

Most operating zinc refineries use precipitation methods for zinc purification, followed by metal recovery by EW. In such processes there usually is a gradual build-up of acid and metals such as zinc and traces of other metals and it is necessary to bleed the electrolyte to control the composition. Several studies have looked at the use of SX for the recovery of both sulphuric acid and zinc from this bleed stream (Buttinelli et al., 1984, 1989). There are several methods to treat acidic effluents such as neutralization, electrolysis solvent extraction etc. Of these neutralization is most widely used method but because of generation of sludge and its disposal causes pollution. Presently

newer challenges for operating metal processing industries to reduce cost and improve product quality while eliminating or minimising waste stream generation, have given an impetus to the development of acid recovery processes and produce value added products. Although most of the zinc refineries recycle the spent electrolyte after makeup of the depleted components and purification with respect to the undesired components, however extraction of sulphuric acid from such solutions and synthesis of the value added product such as zinc oxide can be another environmental friendly option. In this regards acid extraction by several solvents have been tried. Solutions of tertiary amines in organic diluents have been used for separation of the acid from other constituents in an aqueous solution by several workers (Allen, 1956; Ritcey and Ashbrook, 1979; Lassner et al., 1983; McInnis et al., 1983). The extraction equilibria between the aqueous solution of sulphuric acid and tri-*n*-octylamine (TOA) in benzene were measured at 25 °C with varying concentration of amine (Allen, 1956). Acid extraction with Alamine 336 in

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benzene was done by Kim and Chiola (1968), Various aspects such as degree of aggregation of various amines, their sulphates, and uranium sulphates in benzene were also examined by several workers (Allen, 1958; Fomin et al., 1959; Coleman and Roddy, 1966) where they found that there was no aggregation when TOA extracted a normal sulphate where as hydrogen sulphate forms aggregate with average aggregation numbers of 2–3.4.

Extraction of sulfuric acid with tri-*n*-octylamine was also studied in nonvolatile diluents such as dibutyl phthalate, phenylcyclohexane, *n*-hexadecane, along with 5 vol.% of dodecyl alcohol as the modifier (Schmidt, 1980). The data show appreciable differences in the extraction equilibrium with different diluents. Eyal and Canari (1995) found that hydrochloric acid can be strongly extracted by straight-chain aliphatic amines diluted with alcoholic diluent by ion-pair formation. This mechanism may be represented by a two-step process of amine protonation and of anion addition. Earlier Eyal et al. (1993) studied the extraction of acid, water and hydrophilic molecules by amine and amine salts. Acid–base extractants also known as couple extractants have been widely used by, Eyal and Baniel (1984), Eyal et al. (1986) and Eyal (1989). These extractants are composed of amine-organic acid salts in a diluent which is immiscible in water. These extractants show efficient, selective and reversible extraction of mineral acids and their salts.

Solvating reagents such as Cyanex 923 has proved its effectiveness in the separation of hazardous metals and mineral acids from various types of aqueous solutions (Alguacil and Lopez (1996), Alguacil and Martinez (2001), Gupta et al. (2002), Regel et al. (2001), Rickelton (1999), Saji et al. (1998), and Wang et al. (2002)), in which the stripping operation is more easily achieved as compared to that of amine extractants (Regel et al., 2001; Wolters et al., 2002). However a few data are available in the literature about considering liquid–liquid extraction in the treatment of such highly acidic solutions/ waste waters. Hence the aim of this work was to explore the extraction of sulphuric acid from spent zinc bleed stream (SZBS) generated during the electrowinning of zinc in the zinc refineries using dilute TEHA.

## 2. Materials

### 2.1. Sample collection, solutions and reagents

10 L of spent zinc electrolyte was collected from an electroplating industry and the chemical analysis of this waste solution is given in Table 1.

#### 2.1.1. Reagents

The organic reagent used for the recovery of acid in this study was Tris 2-ethylhexyl amine (TEHA) procured from Sigma–Aldrich and was used for the extraction studies without further purification. TEHA is a tertiary amine with a molecular formula as  $[\text{CH}_3(\text{CH}_2)_3\text{CH}(\text{C}_2\text{H}_5)\text{CH}_2]_3\text{N}$ . It is a colourless liquid practically insoluble in water with a molecular weight of 353.7, a density of 0.817 g/mL at 20 °C, and a refractive index of  $n_{20/D}$  1.451. Distilled kerosene

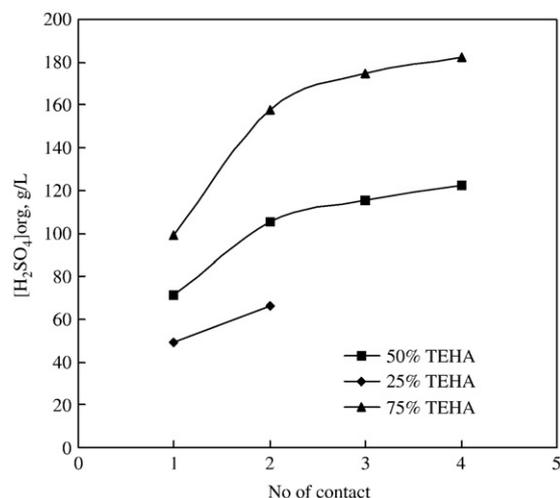


Fig. 1. Effect of TEHA concentration on acid extraction from SZBS.

was used as a diluent to prepare the solvent of different concentration. Effect of other organic solvents used as diluent and phase modifier are: methyl isobutyl ketone (MIBK), tributyl phosphate (TBP), benzene, hexane.

### 2.1.2. Chemical analysis

Chemical analysis of the SZBS before and after acid extraction, in the solutions were done volumetrically by standard methods as mentioned in Vogel (1989) and zinc in the solutions was analysed by 0.1 N ethylenediamine tetra acetic acid (EDTA) in presence of EBT as an indicator at pH 10. Mass balance gives the amount of acid extracted by the solvent. Trace metal analysis was done by atomic absorption spectro-photometer (Thermo SOLAAR S-2).

### 2.2. Experimental

Acid extraction from spent zinc bleed stream (SZBS) was done by 75% TEHA in kerosene. Loading capacity of the solvent for the acid from SZBS containing 173.5 g/L of sulphuric acid and 86.96 g/L of Zn, was performed at 30 °C, 45 °C and 60 °C by repeated contact method at O/A ratio of 1:1. The organic and aqueous solutions were contacted for a period of 5 min to ensure complete extraction in a separatory funnel. The mixture was then left for phase separation and the acid loaded organic phase was separated from the aqueous phase containing un-extracted acid along with the zinc. The same organic was shaken again with the fresh aqueous feed till the organic is completely loaded with the acid, which is indicated by the chemical analysis of the aqueous phase after each contact. Various other parameters such as mixing time, O/A ratio, mixing temperature, aqueous and organic concentration etc. were optimised for maximum acid extraction by TEHA. After the extraction of acid by the organic phase stripping was performed by hot distilled water to recover the loaded acid. The raffinate generated during the extraction stage can be used for the preparation of pure zinc oxide.

## 3. Results and discussion

Sulphuric acid extraction from spent zinc bleed stream containing 173.5 g/L acid and 86.96 g/L Zn along with traces of Ni was performed with different concentration of TEHA.

### 3.1. Rate of extraction

Extraction rate was studied by shaking aqueous phases containing 173.5 g/L acid with the organic phase for different time intervals up to 10 min. The two phases were then allowed to separate and analysed for the amount of acid and zinc taken up by the organic phase. It was found that 5 min of shaking is required for the equilibrium to reach at 30 °C with

Table 1  
Chemical analysis of spent zinc electrolyte

Constituents	H <sub>2</sub> SO <sub>4</sub>	Zn	Ni
Composition	173.5 g/L	86.96 g/L	0.84 ppm

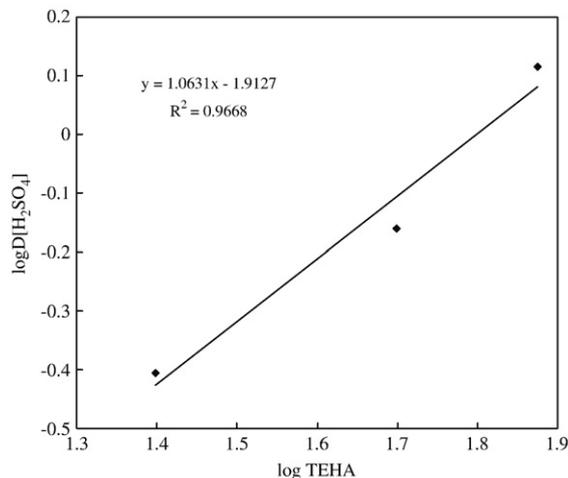


Fig. 2. Plot of  $\log[\text{H}_2\text{SO}_4]$  vs  $\log[\text{TEHA}]$ .

a loading of 56.88% of acid in a single contact. Beyond which the percent extraction remains constant. Extraction of zinc was negligible in sulphate medium. Hence in all the further experiments, shaking time in all the experiments was maintained at 5 min to ensure complete extraction.

### 3.2. Effect of concentration of TEHA

The same aqueous phase was given contact with different concentration of TEHA and the results are represented in Fig. 1. It was observed that percentage extraction of acid is increasing with the increase in the organic concentration. About 1–2% of zinc was also found to be co-extracted with the increase in solvent concentration but this may not be the extracted zinc, it may be the zinc entrapped in the solvent. Probably zinc as sulphate has a very less affinity for TEHA. However presence of chloride in the system shows the extraction of zinc as its chloro complex by this solvent. Hence zinc could also be extracted from the raffinate by adding chloride ions to the system. Thus with the increase in solvent concentration from 25% to 75% the uptake of acid increases from 49 g/L to 99 g/L in 1st contact and the saturated loading capacity was found to be attained in 4 contacts with the same organic feed and fresh aqueous feed at an O/A ratio of 1, with a shaking time of 5 min at 30 °C. The saturated loading capacity was found to increase from 66 g/L for 25% TEHA to 122 g/L for 50% solvent and 182 g/L for 75% solvent. Also in case of 25% TEHA, saturation was reached in two contacts and there was no extraction on further increase

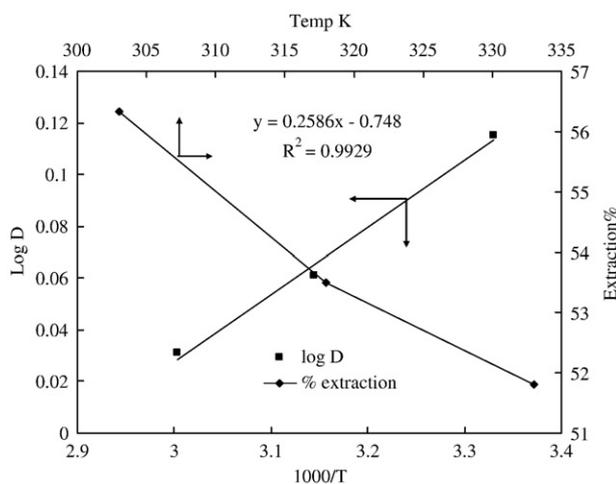


Fig. 3. Effect of temperature on extraction of acid by TEHA.

Table 2

Thermodynamic functions for the extraction of  $\text{H}_2\text{SO}_4$  from spent zinc bleed stream by 75% TEHA

Temp K	$\Delta G/\text{kJ mol}^{-1}$	$\Delta S/\text{kJ mol}^{-1} \text{K}^{-1}$	$\Delta H/\text{kJ mol}^{-1}$
303	-0.646	-14.2	-4.95
318	-0.372	-14.4	
333	-0.199	-14.27	

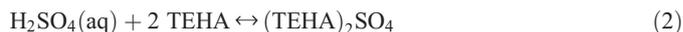
in contact. Therefore with the aim to get maximum acid extraction and to get a concentrated regenerated acid, further experiments were performed with 75% TEHA. It was also found that as the TEHA concentration increase, the solvent becomes viscous after the loading of acid but the phase separation was clear. This acid loaded organic was then treated with hot distilled water to strip out the acid and TEHA was regenerated in its original form without affecting its extraction property. Plot of  $\log[\text{H}_2\text{SO}_4]$  vs  $\log[\text{TEHA}]$  shows a straight line with slope equal to 1.06 and  $R$  value of 0.9668 showing that 1 mole of sulphuric acid is extracted by 1 mol of TEHA (Fig. 2).

Thus the extraction of sulphuric acid by TEHA can be written as:

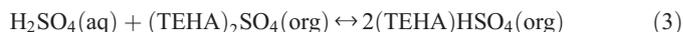


where  $x = 1$ .

Similar observations were made by Gottlieb et al. (2000) where they have shown that when the acid concentration in the aqueous solution is below 1 M, the reaction between TEHA and  $\text{H}_2\text{SO}_4$  proceeds by the formation of amine sulphate where 2 molecules of TEHA combined with one  $\text{H}_2\text{SO}_4$ . This can be represented as



and when the acid concentration is  $> 1 \text{ M}$ , the final product is amine bisulphate as represented below.



### 3.3. Effect of temperature on the extraction of acid from the SZBS

Acid extraction experiments were performed at different temperatures ranging from 30 to 60 °C from an aqueous feed containing 173.5 g/L acid and 86.96 g/L of zinc with 75% TEHA. Results are represented in Fig. 3. With increase in temperature from 30 °C to 60 °C, the percentage extraction decreases from 56.8 to 51.4%. So all the further experiments

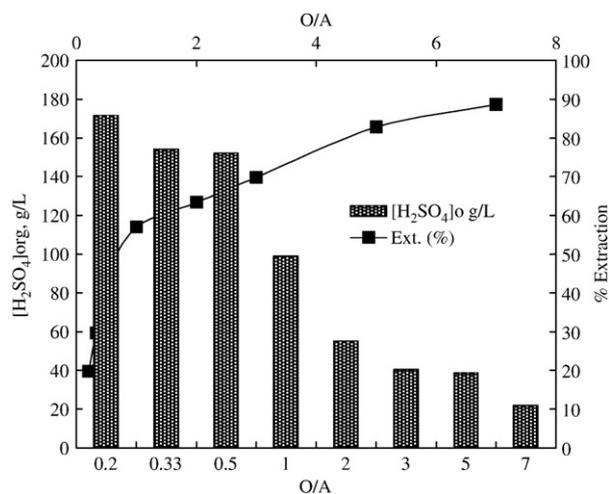


Fig. 4. Effect of O/A on acid extraction by TEHA.

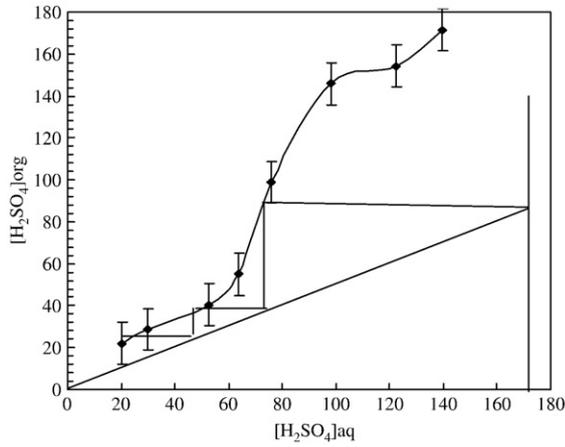


Fig. 5. McCabe Thiele plot for the extraction of acid from SZBS by TEHA.

were performed at 30 °C for the extraction studies. Effect of temperature can be better understood in terms of thermodynamic calculations. Hence a plot of  $1000/T$  vs  $\log D_{[H_2SO_4]}$  Fig. 3 gives a straight with a slope of 0.2586 and  $R$  value of 0.9929. Thus change in enthalpy of the extraction processes,  $\Delta H$ , can be obtained from the slope of  $\log D$  vs  $1/T$  using the Van't Hoff Equation (Sun et al., 2007):

$$\log D = -\frac{\Delta H}{2.303 RT} + C \quad (4)$$

Where  $R$  is the gas constant and  $C$  is integration constant which includes the equilibrium constant of the extraction reaction and the activity coefficients for other components, which are assumed to be invariant under the experimental conditions. Thus  $\Delta H$  value was calculated to be  $-4.95 \text{ kJmol}^{-1}$ . A negative value of  $\Delta H$  indicated the extraction to be exothermic in nature hence an increase in temperature decreases the up take of acid by the solvent. Further the free energy  $\Delta G$  and the entropy were calculated from the following equations

$$\Delta G = -RT \ln D = -2.303 RT \log D \quad (5)$$

and

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (6)$$

Thus the thermodynamic parameters given in Table 2 were evaluated using Eqs. (5) and (6). The negative free energy value shows that

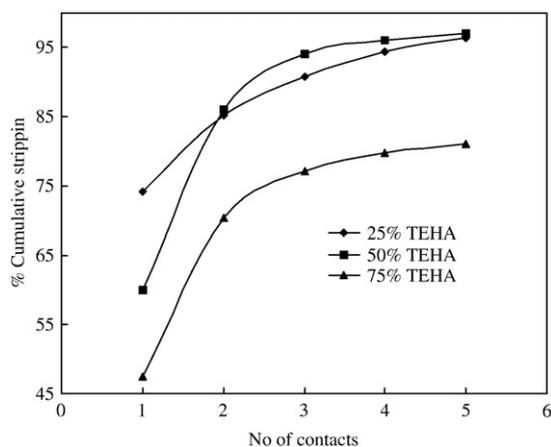


Fig. 6. Stripping of loaded acid from the acid loaded TEHA of different concentration.

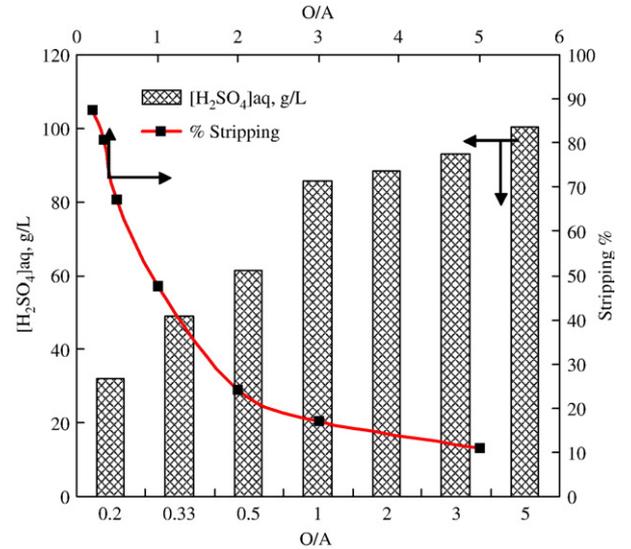


Fig. 7. Effect of O/A on the stripping of loaded acid from 75% TEHA with hot distilled water.

acid extraction from spent zinc bleed stream by TEHA is thermodynamically favourable. The negative  $\Delta S$  values obtained from the investigation shows that more order is introduced in the system on acid extraction.

### 3.4. Effect of O/A on the extraction of acid with TEHA

To extract maximum amount of acid from SZBS, the aqueous feed was contacted with the organic feed in different ratio. Fig. 4 shows that with the increase in O/A ratio the percentage acid extraction increases, viz. acid extraction increases from 19.7% to 94% with increase in O/A ratio from 0.2 to 5 at 30 °C. McCabe thiele plot (Fig. 5) for the extraction of acid at different O/A ratio with 75% TEHA and SZBS shows that at O/A ratio of 2 the amount of acid can be brought down from 173.9 g/L to about 20 g/L in three contacts. Raffinate containing about 20 g/L of acid and all the zinc can be directly used for the synthesis of value added products such as ZnO directly.

Thus, TEHA was found to be a very effective extractant for extraction of sulphuric acid from a source containing very high acid. Therefore,

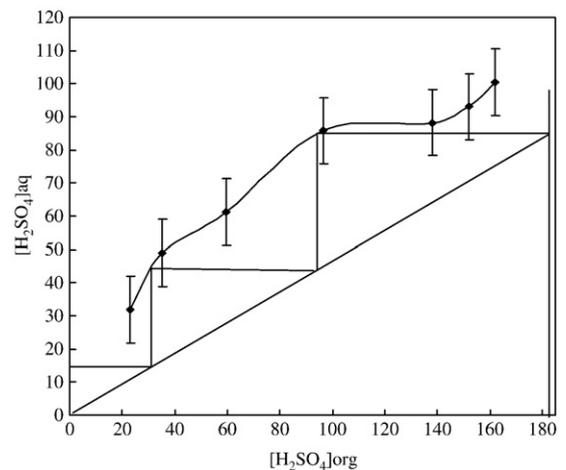


Fig. 8. McCabe Thiele plot for the stripping of loaded acid from 75% TEHA ( $[H_2SO_4]$  org).

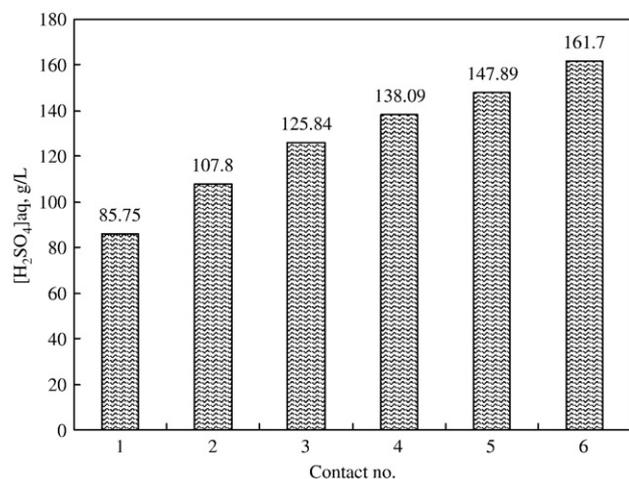


Fig. 9. Stripping of loaded acid by repeated contact of same aqueous feed with fresh loaded organic.

a detailed stripping study was carried out for its application for acid extraction.

### 3.5. Effect of time on the stripping of acid with fresh distilled water in each contact

Stripping experiments were carried out using the acid loaded organic with 182 g/L acid, at different time interval with distilled water at 60 °C. Percentage stripping of acid increases with time and becomes maximum in 5 min, beyond which it remains constant. Hence in all the stripping experiments, loaded organic and hot distilled water were shaken for 5 min. Since during extraction, the loading of acid decreases with temperature, therefore the stripping studies were performed at 60 °C.

### 3.6. Stripping of loaded acid from solvent of different concentration

Stripping of acid from the acid loaded solvents of different concentration was carried out with distilled water and is represented in Fig. 6. Almost 99% of acid was stripped from the loaded organic and percentage of acid stripping increases marginally with increasing the concentration of loaded solvent. The total acid stripped in four contacts increased from 97% to 99% with increasing solvent (TEHA) concentration from 25% to 75%. Analysis of zinc content shows that about 1.7 g/L of zinc is stripped which was co-extracted with the acid.

### 3.7. Effect of O/A on stripping of loaded acid from TEHA

Stripping experiments were performed at different O/A ratio. Plot of O/A vs H<sub>2</sub>SO<sub>4</sub> stripped in g/L and O/A vs percentage stripping at 60 °C were represented in Fig. 7. It can be seen from the plot that with the increase in the O/A ratio from 0.2 to 5.0, the concentration of stripped acid increases from 31.9 to 100.9 g/L but the percent stripping decreases from 87% to 11. McCabe Thiele plot (Fig. 8) for the stripping of loaded acid shows the requirement of three stages at O/A ratio of 1: 2.11.

### 3.8. Stripping of loaded acid by repeat contact method with fresh loaded organic

To get a concentrated acid which could be recycled back in to the system, same aqueous feed was contacted with the fresh loaded organic. The concentration of stripped acid increases with increase in the number of contacts and about 161.7 g/L acid is regenerated in 6 contacts (Fig. 9) at

an O/A ratio of 1:1. However, the percent acid stripped decreases in each contact and about 54.5% of the loaded acid is stripped in first contact.

## 4. Conclusions

Solvent extraction of sulfuric acid from the spent zinc bleed stream was carried out using TEHA. The extraction of H<sub>2</sub>SO<sub>4</sub> increased with the increase of solvent concentration. There was no extraction of zinc in dilute TEHA solution, however with the increase in TEHA concentration to 75% about 1–2% of zinc was found to be extracted. The log D vs. log [TEHA] was a straight line with slope values of 1.063 indicating that 1 mol of the TEHA was involved with 1 mol of the extracted acid. McCabe Thiele construction indicated the possibility of about 90% H<sub>2</sub>SO<sub>4</sub> extraction in three counter-current stages at the A:O of 1:2. Loaded acid was stripped with hot distilled water at 60 °C. McCabe Thiele plot shows that at O/A of 1: 2.11, the loaded acid was stripped in three stages. Hence TEHA is a prospective reagent for the extraction of sulphuric acid from the industrial waste streams such as spent zinc bleed stream of a zinc electroplating industry leaving behind zinc which can be used for the preparation of pure zinc oxide. This work has been performed on a bench scale and to develop a complete technology some pilot scale experiments need to be done.

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